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In situ Femtosecond Laser and Argon Ion Beams for 3D Microanalysis using the TriBeam

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Advanced engineering materials require microstructural characterization in 3D across lengthscales, motivating the development of new tomography techniques and coupling with existing capabilities. The acquisition of 3D datasets with structural and chemical information at lengthscales between those accessible using Ga^+ and Xeon FIB SEMs and those of X-ray tomography techniques is still challenging, particularly for dense multiphase materials. Femtosecond lasers have been employed for low damage [1,2] material removal, in tomography applications, over mm^3 regions *in situ* in a FIB SEM as shown in Figure 1. FIB cross sections investigated by TEM have shown that dislocations can be injected to microns in depth in some materials [3], but are primarily confined to less than 100s of nanometers of the surface in the low fluence ablation regime. Parametric studies of laser fluence and beam scanning conditions in silicon in the TriBeam show that, when the propagating laser beam is scanned parallel with the sample surface, the damage is exclusively limited to that of the low fluence ablation regime. Laser ablation studies have also shown the ability to resolve surface sensitive EBSD maps from the ablated surface of many metals and/or alloys primarily containing magnesium, titanium, nickel, steel, copper, tungsten, tin and niobium.

Recently, a broad beam argon ion source capable of microamp currents has been integrated into the TriBeam for the further reduction in surface damage by glancing angle milling. Previous *in situ* Ar^+ ion beam experiments show ion induced amorphization in Si can be reduced to 10s of nm at low accelerating voltages [4]. Femtosecond laser ablated silicon samples were investigated, as well as samples that had been subsequently Ga^+ ion milled, as shown in Figure 1 (right). TEM lamellae were extracted to determine the changes in surface structure and amorphization depths as a function of types of beam exposure. A brightfield TEM image in Figure 2 demonstrates amorphization depths of 20 nm as resulting from laser machining with fluences at 20x the ablation threshold, which were scanned in a surface parallel to the beam orientation.

Currently an optimal balance for microanalaytical 3D dataset acquisition (EBSD and EDS), dictated by detector speed and sensitivity, must be struck between in plane resolution and experiment duration. Enhancements in 3D EBSD collection speed and pattern quality will be discussed for femtosecond laser ablated and Ar^+ ion cleaned surfaces in the context of multimodal data collection of NiTiSn alloys for thermoelectric applications.

References:

[1] S. Ma *et al*, Met. Mat. Trans. A **38** (2007) p. 2349-2357.
[2] Q. Feng *et al*, Scripta Mat. **53**(5) (2005) p. 511-516.
[3] M.P. Echlin *et al*, Materials Characterization: Tutorial Review **100** (2015) p. 1-12.
[4] L.A. Giannuzzi *et al*, Microscopy and Microanalysis **11** (2005) p. 828-829.

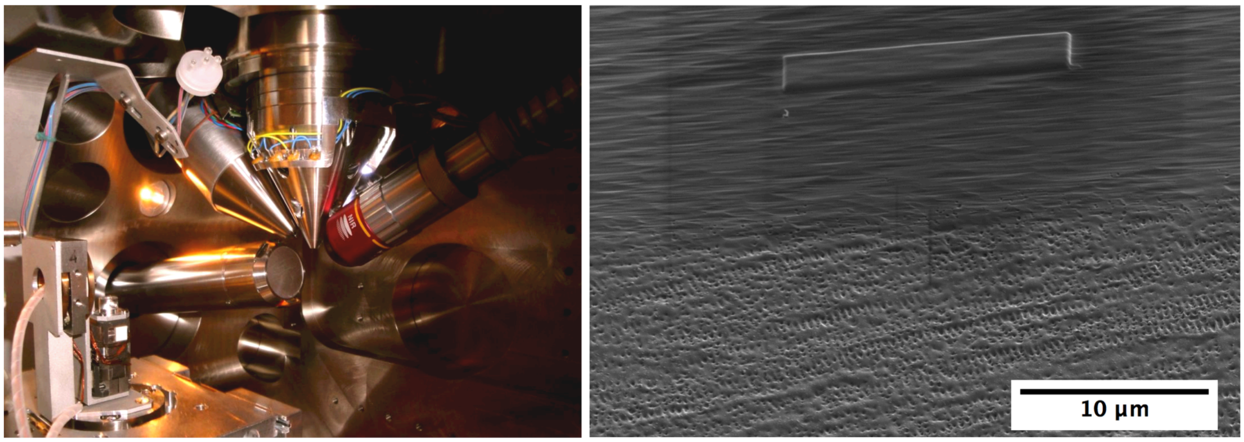

Figure 1. (left) The TriBeam tomography FIB-SEM vacuum chamber interior. (right) A silicon sample that was femtosecond laser ablated and then Ga^+ ion milled at glancing angle (top half of micrograph). The location of a TEM lamella liftout is visible as a raised platinum deposited area.

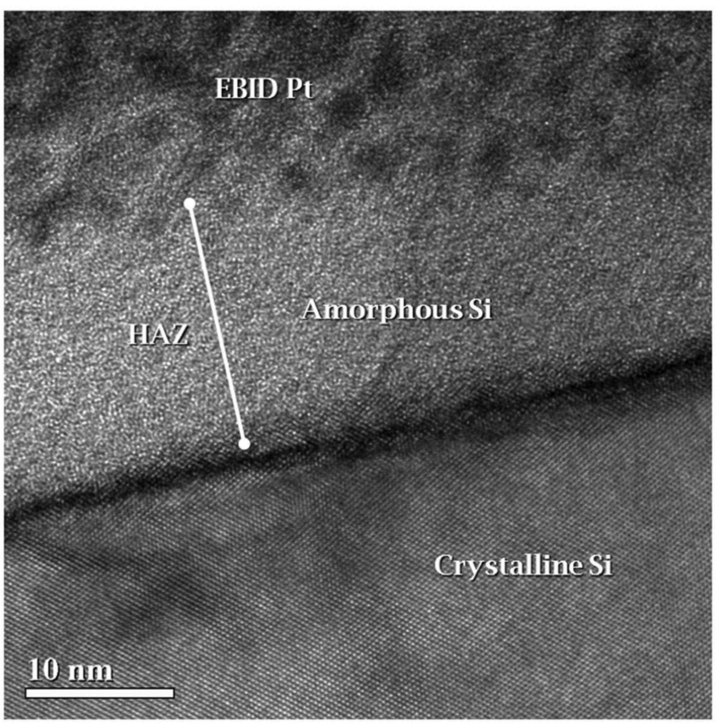

Figure 2. A brightfield TEM image of a lamella extracted from a silicon sample that was femtosecond laser machined at a glancing angle showing approximately 20nm of amorphization depth.